PNWD-2466
BNFL-RPT-004, Rev. 0

LETTER REPORT

AW-101 Entrained Solids – Solubility Versus Temperature

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Project 29952/29953
Battelle, Richland, Washington, 99352

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Contents

Introduction

BNFL, Inc. (BNFL) is under contract with the U.S. Department of Energy, River Protection Project (DOE-RPP) to design, construct, and operate facilities for the treatment of wastes stored in the single-shell and double-shell tanks at the Hanford Site, Richland Washington. BNFL has contracted with Battelle Pacific Northwest Division to conduct tests to verify and validate the BNFL waste treatment process. The DOE-RPP has provided samples from tanks 241-AW-101, 241-AN-107, 241-C-104, and 241-C-106 to BNFL for this purpose.

This report describes the results of a test conducted by Battelle to assess the solubility of the solids entrained in the diluted AW-101 low-activity waste (LAW) sample. BNFL requested Battelle to dilute the AW-101 sample using de-ionized water to mimic expected plant operating conditions. BNFL further requested Battelle to assess the solubility of the solids present in the diluted AW-101 sample versus temperature conditions of 30, 40, and 50°C. BNFL requested these tests to assess the composition of the LAW supernatant and solids versus expected plant-operating conditions. The work was conducted according to test plan BNFL-TP-29953-7, Rev. 0, *Determination of the Solubility of LAW Entrained Solids*. The test went according to plan, with no deviations from the test plan.

Personnel

The Battelle personnel and their responsibilities in performing this test are given below.

Staff Member	Responsibilities
G.J. Lumetta	Cognizant scientist. Prepared test plan and designed experiment. Supervised performance of the test. Prepared analytical service request. Interpreted data and reported results.
R.C. Lettau	Hot cell technician. Performed test.
M.W. Urie	Managed chemical and radiochemical analytical work.
B.M. Rapko	Technical reviewer.
K.P. Brooks	Task Leader.
G.F. Piepel	Statistical analysis of the data.

Experimental

Sample Description. The sample used in this test was labeled as AW-101 ST. The homogenization, dilution, caustic adjustment, and sub-sampling of the as-received AW-101 sample were described by Urie 1999. The total volume of sample AW-101 ST was 25 mL and it contained approximately 2 mL of settled solids.

Apparatus. The apparatus used consisted of an aluminum heating block placed on a hot plate/stirrer. The hot plate/stirrer was modified so that separate power could be applied to the heating and stirring functions. This allowed for continuous stirring, while the hot plate was powered by a temperature controller. The temperature controller used was a J-KEM Model 270 (J-KEM

Electronics, Inc., St. Louis, MO). This temperature controller consists of two separate circuits. One is the temperature control circuit, while the other serves as an over-temperature device, which shuts down the system if a preset temperature is exceeded. The set point for the over-temperature circuit was set at 60°C for this test. A dual K-type thermocouple (model number CASS-116G-12-DUAL, Omega Engineering, Stamford, CT) was used to provide inputs to the temperature controller and over-temperature circuits. Both the J-KEM Model 270 and the dual thermocouple were calibrated before use. The aluminum heating block contained two wells. A vial containing water was placed in one of the wells, with the thermocouple wedged between this vial and the aluminum block. The vial containing the sample was placed in the other well.

Procedure.[a] The sample in AW-101 ST was mixed by swirling. The homogenized slurry was then transferred to a 30-mL high density polyethylene (HDPE) vial (this vial also contained a Teflon®-coated magnetic stir bar). The sample was heated and stirred at 30 ± 2 °C for 1.5 h. Two aliquots (2-mL each) were taken for analysis. Each aliquot was immediately filtered through a 0.45-µm nylon syringe filter that had been preheated by immersion in a boiling water bath. The filter was preheated to avoid precipitation during the filtration step. The temperature was increased to 40 ± 2 °C and the sample was stirred for 16.75 h.[b] The mixture was sampled in the same manner as described above. The temperature was increased to 50 ± 2 °C and the sample was stirred for 1.25 h. Again, the mixture was sampled in the same manner as described above. The filtered samples were subjected to the following analytical procedures: IC(anions), TOC/TIC, acid digestion, ICP/AES, ICP-MS(Tc-99), Sr-90, total alpha, total uranium, and GEA.

Results

Tables 1, 2, and 3 present the concentrations of various waste components at 30, 40, and 50°C, respectively. Table 4 shows the changes in the concentrations at 40 and 50°C relative to those at 30°C. Appendix D discusses a graphical analysis of the data, as well as linear regression results of fitting component concentrations versus temperature. The following discussion is organized according to the following types of components: 1) radionuclides, 2) bulk metals and carbon, and 3) anions.

Radionuclides. The data suggest that the ^{137}Cs concentration increased slightly with temperature. Increases of 2.3 and 5.6% in the ^{137}Cs concentration occurred in going from 30 to 40°C and from 30 to 50°C, respectively. Nearly identical increases were seen in the ^{99}Tc concentrations. The increases in ^{137}Cs and ^{99}Tc concentrations from 30 to 50°C were statistically significant (see Table 4). Linear regressions of ^{137}Cs and ^{99}Tc concentrations versus temperature had statistically significant positive slopes (see Appendix D).

On the other hand, the ^{90}Sr concentrations appeared to decrease with increasing temperature. However there is considerable scatter in the ^{90}Sr data; the standard deviations range from 20 – 58% of the mean ^{90}Sr concentration values at each temperature. Thus, the indicated changes in the ^{90}Sr

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[a] The test plan and the associated procedural notes are included as Appendix A to this report.

[b] The test plan required the AW-101 sample to be maintained at temperature for at least 1 hour before sampling. For convenience, the sample was maintained at 40°C overnight. It should be noted that this test was not designed to address the kinetics of dissolution. Kinetics could potentially be important regarding the phenomena investigated here, but separate testing would be required to address this issue.

concentrations are not statistically significant (see Table 4). Similarly, the linear regression of ^{90}Sr concentration versus temperature had a slope that was not statistically different from zero (see Appendix D). The reason for the relatively high uncertainty in ^{90}Sr concentrations was the relatively high background caused by the ^{85}Sr tracer that was added in the analytical procedure to monitor Sr recovery.

All the transuranic elements (alpha emitters) were below the detection limits. Likewise, the europium isotopes $^{154,155}Eu$ were not detected. It should be noted that the detection limits for the Eu isotopes were somewhat high because of the strong ^{137}Cs activity in the sample.

Bulk Metals and Carbon. Most of the metals analyzed showed slight concentration increases with increasing temperature. Most notable are the increases seen for Al, Cr, and U. Increases in these three components are statistically significant based on the analyses presented in Table 4 and Appendix D. The Fe concentration increased approximately 20% when the temperature was raised to 40 or 50°C, with the increases assessed to be statistically significant (see Table 4). However, Fe was near the detection limit so that there is significant experimental uncertainty associated with this result. The concentrations of K, Na, Ni, P, and Zr also displayed statistically significantly increases with increasing temperature.

The total organic carbon (TOC) concentration in the AW-101 solution also increased slightly with increasing temperature, with the increase being statistically significant (see Table 4 and Appendix D). The average total inorganic carbon (TIC) concentration increased when the temperature was raised from 30 to 40°C, but the TIC concentration did not increase further when the temperature was raised to 50°C. The changes from 30 to 40°C and 30 to 50°C are not statistically significant, because of the experimental uncertainties in the TIC measurements (see Table 4).

Anions. The data suggest that the F^- concentration increased with temperature, with the increase being statistically significant. Increases of 12.5 and 19.2% in the F^- concentration occurred in going from 30 to 40°C and from 30 to 50°C, respectively (see Table 4). The linear regression of F^- concentration versus temperature also had a statistically significant positive slope (see Appendix D). The data also suggest the average Cl^- concentration increased when the temperature was raised from 30 to 40°C, although the increase was not statistically significant (see Table 4). The Cl^- concentration did not increase further when the temperature was raised to 50°C. Statistical analyses of these data suggest the F^- and Cl^- concentration increases should be considered with caution as there is considerable scatter in the data. Sulfate and phosphate ions were below the detection limits of the ion chromatograph. Assuming phosphate ion is the dominant form of P in solution, the behavior of PO_4^{3-} can be deduced from the ICP data as discussed above.

In determining the concentration of NO_3^-, there was a significant discrepancy between the duplicate analyses for the solution taken at 40°C. In particular, the nitrate concentration value obtained for sample AW101-SOL-40A2 was about twice that obtained for sample AW101-SOL-40A1. Furthermore, the value of 131,250 μg/mL obtained for sample AW101-SOL-40A1 was more in line with those obtained for the solution at 30 and 50°C. This strongly suggests that the value reported for AW101-SOL-40A2 is in error. Perhaps this error was caused by nitrate contamination of the sample, or a dilution error. Using the value of 131,250 μg/mL at 40°C, the data suggest a 10% increase in the nitrate concentration when the temperature is raised from 30 to 40°C. However, there was a decrease in the nitrate concentration when the temperature was raised from 40 to 50°C. The linear regression of NO_3^- concentration versus temperature (omitting the concentration value

for AW101-SOL-40A2 as an outlier) had a statistically significant lack of fit (see Appendix D). This result suggests that a quadratic rather than linear relationship may be more appropriate. However, the limited nature of the data (especially after omitting the outlier) raises the question whether the decrease between 40 and 50°C is significant.

Conclusions

The data are limited because they are based on a single AW-101 sample, from which was obtained two subsamples/analyses at each of three temperatures. Further, the data for some AW-101 components are subject to considerable uncertainty. However, there does appear to be an overall trend for the concentrations of certain AW-101 waste components (e.g., ^{137}Cs, ^{99}Tc, Al, Cr, K, Na, Ni, P, U, Zr, TOC, F^-, and NO_3^-) to increase with increasing temperature. Typical increases were on the order of 2 to 5% for each 10°C increase, although a fewer larger increases were seen for some components. Because the sample bottle was sealed during the course of the experiment, evaporation in not a likely cause for the observed concentration increases.

Reference

Urie, M.W. et al. 1999. *Inorganic and Radiochemical Analysis of AW-10 and AN-107 "Diluted Feed" Materials*, PNWD-2463, Battelle Pacific Northwest Division, Richland, Washington.

Table 1. AW-101 Component Concentrations in Solution at 30°C.[a]

Analyte	Concentration at 30°C			Std. Dev.
	AW101-SOL-30A1	AW101-SOL-30A2	Mean	
Cesium-137	255	264	260	6
Strontium-90	0.949	0.400	0.675	0.388
Technetium-99	0.103	0.106	0.104	0.002
Americium-241	< 6E-03	< 6E-03	< 6E-03	--
Europium-154	< 1E-02	< 9E-03	< 9E-03	--
Europium-155	< 2E-01	< 2E-01	< 2E-01	--
Total Alpha	< 6E-03	< 6E-03	< 6E-03	--
Ag	(0.81)	(0.77)	(0.79)	0.03
Al	17600	18300	17950	495
Ba	< 5.0	< 5.0	< 5.0	--
Ca	(9.3)	(11.0)	(10.2)	1.2
Cd	(2.1)	(2.0)	(2.1)	0.1
Co	< 12.5	< 12.5	< 12.5	--
Cr	62.9	65.0	64.0	1.5
Cu	(1.6)	(1.6)	(1.6)	0.0
Fe[b]	(3.5)	(3.5)	(3.5)	0.0
K	24400	25600	25000	849
La	< 12.5	< 12.5	< 12.5	--
Mg	< 50	< 50	< 50	--
Mn	< 2.5	< 2.5	< 2.5	--
Mo	< 15	< 15	< 15	--
Na	143000	145000	144000	1414
Ni	(5.1)	(5.3)	(5.2)	0.1
P	344	358	351	10
Pb	38.9	42.7	40.8	2.7
Si[c]	264	202	233	44
Ti	< 2.5	< 2.5	< 2.5	--
U	2.73	2.80	2.77	0.05
Zn[d]	(6.3)	(6.7)	(6.5)	0.3
Zr	(6.7)	(6.8)	(6.8)	0.1
TOC	1900	1940	1920	28
TIC	2760	2960	2860	141
Cl^-	3600	4100	3850	354
F^-	1300	1300	1300	0
NO_3^-	118000	120000	119000	1414
SO_4^{2-}	< 1200	< 1000	< 1200	--
PO_4^{3-}	< 1200	< 1000	< 1200	--

(a) Concentrations for radionuclides are in units of µCi/mL; all other components are in units of µg/mL. Values in parentheses are near the analytical detection limit.
(b) The process blank had a relatively high Fe content of 0.4 µg/mL.
(c) The process blank had a relatively high Si content of 119 µg/mL.
(d) The process blank had a relatively high Zn content of 0.3 µg/mL.

Table 2. AW-101 Component Concentrations in Solution at 40°C.[a]

Analyte	Concentration at 40°C			
	AW101-SOL-40A1	AW101-SOL-40A2	Mean	Std. Dev.
Cesium-137	267	264	266	2
Strontium-90	0.519	0.696	0.608	0.125
Technetium-99	0.107	0.106	0.107	0.001
Americium-241	< 7E-03	< 6E-03	< 6E-03	--
Europium-154	< 1E-02	< 1E-02	< 1E-02	--
Europium-155	< 2E-02	< 2E-02	< 2E-02	--
Total Alpha	< 7E-03	< 6E-03	< 6E-03	--
Ag	(0.84)	(0.81)	(0.83)	0.02
Al	18600	18600	18600	0
Ba	< 5.0	< 5.0	< 5.0	--
Ca	(11.0)	(11.0)	(11.0)	0.0
Cd	(2.1)	(2.0)	(2.1)	0.1
Co	< 12.5	< 12.5	< 12.5	--
Cr	67.5	67.4	67.5	0.1
Cu	(1.6)	(1.5)	(1.6)	0.1
Fe[b]	(4.4)	(4.0)	(4.2)	0.3
K	26000	26000	26000	0
La	< 12.5	< 12.5	< 12.5	--
Mg	< 50	< 50	< 50	--
Mn	< 2.5	< 2.5	< 2.5	--
Mo	< 15	< 15	< 15	--
Na	146000	146000	146000	0
Ni	(5.3)	(5.2)	(5.3)	0.1
P	361	357	359	3
Pb	48.6	40	44.3	6.1
Si[c]	269	274	272	4
Ti	< 2.5	< 2.5	< 2.5	--
U	3.00	2.98	2.99	0.01
Zn[d]	(6.6)	(6.6)	(6.6)	0.0
Zr	(7.0)	(7.0)	(7.0)	0.0
TOC	2010	1960	1985	35
TIC	3040	2940	2990	71
Cl^-	4050	4200	4125	106
F^-	1325	1600	1462.5	194
NO_3^-	131250	227000	179125	67705
SO_4^{2-}	< 1600	< 1200	< 1600	--
PO_4^{3-}	< 1400	< 1200	< 1400	--

(a) Concentrations for radionuclides are in units of µCi/mL; all other components are in units of µg/mL. Values in parentheses are within 10 times the analytical detection limit.
(b) The process blank had a relatively high Fe content of 0.4 µg/mL.
(c) The process blank had a relatively high Si content of 119 µg/mL.
(d) The process blank had a relatively high Zn content of 0.3 µg/mL.

Table 3. AW-101 Component Concentrations in Solution at 50°C.[a]

Analyte	Concentration at 50°C			Std. Dev.
	AW101-SOL-50A1	AW101-SOL-50A2	Mean	
Cesium-137	276	272	274	3
Strontium-90	0.534	0.352	0.443	0.129
Technetium-99	0.111	0.109	0.110	0.001
Americium-241	< 6E-03	< 8E-03	< 6E-03	--
Europium-154	< 1E-02	< 9E-03	< 9E-03	--
Europium-155	< 7E-02	< 7E-02	< 7E-02	--
Total Alpha	< 6E-03	< 8E-03	< 6E-03	--
Ag	(0.84)	(0.82)	(0.83)	0.01
Al	19200	18700	18950	354
Ba	< 5.0	< 5.0	< 5.0	--
Ca	(12.0)	(10.0)	(11.0)	1.4
Cd	(2.1)	(2.0)	(2.1)	0.1
Co	< 12.5	< 12.5	< 12.5	--
Cr	70.6	68.8	69.7	1.3
Cu	(1.2)	(1.5)	(1.4)	0.2
Fe[b]	(4.4)	(4.2)	(4.3)	0.1
K	26700	26000	26350	495
La	< 12.5	< 12.5	< 12.5	--
Mg	< 50	< 50	< 50	--
Mn	< 2.5	< 2.5	< 2.5	--
Mo	< 15	< 15	< 15	--
Na	147000	146000	146500	707
Ni	(5.5)	(5.5)	(5.5)	0.0
P	372	364	368	6
Pb	40.7	42.0	41.4	0.9
Si[c]	248	269	259	15
Ti	< 2.5	< 2.5	< 2.5	--
U	3.15	3.08	3.12	0.05
Zn[d]	(7.0)	(6.7)	(6.9)	0.2
Zr	(7.2)	(7.1)	(7.2)	0.1
TOC	2010	2030	2020	14
TIC	3170	2730	2950	311
Cl^-	4100	4100	4100	0
F^-	1600	1500	1550	71
NO_3^-	126000	122000	124000	2828
SO_4^{2-}	< 1000	< 1000	< 1000	--
PO_4^{3-}	< 1000	< 1000	< 1000	--

(a) Concentrations for radionuclides are in units of µCi/mL; all other components are in units of µg/mL. Values in parentheses are within 10 times the analytical detection limit.
(b) The process blank had a relatively high Fe content of 0.4 µg/mL.
(c) The process blank had a relatively high Si content of 119 µg/mL.
(d) The process blank had a relatively high Zn content of 0.3 µg/mL.

Table 4. Concentration Changes Relative to 30°C[a]

Analyte	Change, %[b] 40°C	Change, %[b] 50°C	%RSD[c]	SD of % Change[c]	%Change / SD of % Change[c] 40°C	%Change / SD of % Change[c] 50°C
Cesium-137	2.3	**5.6**	1.6	2.3	1.02	**2.46**
Strontium-90	-9.9	-34.3	39.1	55.3	-0.18	-0.62
Technetium-99	2.4	**5.5**	1.4	2.0	1.20	**2.69**
Americium-241	(d)	(d)	(d)	(d)	(d)	(d)
Europium-154	(d)	(d)	(d)	(d)	(d)	(d)
Europium-155	(d)	(d)	(d)	(d)	(d)	(d)
Total Alpha	(d)	(d)	(d)	(d)	(d)	(d)
Ag	(4.4)	(5.1)	2.7	3.9	1.15	1.31
Al	3.6	**5.6**	1.9	2.7	1.33	**2.05**
Ba	(d)	(d)	(d)	(d)	(d)	(d)
Ca	8.4	8.4	10.1	14.3	0.59	0.59
Cd	(0.0)	(0.0)	3.4	4.9	0.00	0.00
Co	(d)	(d)	(d)	(d)	(d)	(d)
Cr	**5.5**	**9.0**	1.7	2.4	**2.27**	**3.73**
Cu	-(3.1)	-(15.6)	9.4	13.4	-0.23	-1.17
Fe	**(20.0)**	**(22.9)**	4.3	6.1	**3.27**	**3.74**
K	4.0	**5.4**	2.2	3.2	1.26	**1.70**
La	(d)	(d)	(d)	(d)	(d)	(d)
Mg	(d)	(d)	(d)	(d)	(d)	(d)
Mn	(d)	(d)	(d)	(d)	(d)	(d)
Mo	(d)	(d)	(d)	(d)	(d)	(d)
Na	1.4	**1.7**	0.6	0.9	1.55	**1.94**
Ni	(1.0)	**(5.8)**	1.8	2.5	0.39	**2.33**
P	2.3	**4.8**	1.9	2.7	0.84	**1.79**
Pb	8.6	1.3	8.9	12.6	0.68	0.11
Si[e]	16.5	10.9	11.4	16.1	1.03	0.68
Ti	(d)	(d)	(d)	(d)	(d)	(d)
U	**8.1**	**12.7**	1.4	2.0	**4.08**	**6.35**
Zn	(1.5)	(5.4)	3.1	4.4	0.35	1.23
Zr	**(3.7)**	**(5.9)**	0.8	1.2	**3.15**	**5.04**
TOC	**3.4**	**5.2**	1.4	2.0	**1.72**	**2.64**
TIC	4.5	3.1	6.9	9.7	0.47	0.32
Cl^-	7.1	6.5	5.5	7.8	0.92	0.83
F^-	12.5	**19.2**	8.1	11.5	1.09	**1.68**
NO_3^-	**10.3**[f]	4.2	1.82[f]	2.57[f]	**4.01**[f]	1.63[f]
SO_4^{2-}	(d)	(d)	(d)	(d)	(d)	(d)
PO_4^{3-}	(d)	(d)	(d)	(d)	(d)	(d)

(a) Values in parentheses are for analytes that were within 10 times the analytical detection limit.

(b) The percent change is given by: $\%Change = 100*(C_T - C_{30})/C_{30}$, where C_T is the average concentration at temperature T (40 or 50°C) and C_{30} is the average concentration at 30°C.

(c) %RSD is the percent relative standard deviation, obtained as the root mean square of the %RSD values at 30°C, 40°C, and 50°C. SD of % Change is the standard deviation of the % Change values at 40°C and 50°C, both relative to 30°C. It is computed as Sqrt(2)*%RSD. % Change/SD of % Change is the number of standard deviations the % Change value is from zero. Assuming a statistical t-distribution with 3 degrees of freedom, % Change/SD of % Change values must be larger than 1.64 to be significant at the 90% (one-sided) confidence level. Such values, and their corresponding % Change values, are shown in boldface.

(d) Analyte not detected.

(e) The values for Si should be veiwed with caution because of the high process blank.

(f) Value obtained using a value of 131250 µg NO_3^-/mL at 40°C.

Appendix A. Test Plan

Appendix B. Raw Data

Appendix C. Calculations

Appendix D. Statistical Analysis of the Data

Appendix A. Test Plan

Work Place Copy

PNNL Test Plan	Document No.: BNFL-TP-29953-7 Rev. No.: 0

Title: Determination of the Solubility of LAW Entrained Solids

Work Location: RPL/SAL	Page 1 of 5
Author: GJ Lumetta	Effective Date: December 14, 1998 Supersedes Date: New
Use Category Identification: Mandatory	
Identified Hazards: __ Radiological __ Hazardous Materials __ Physical Hazards __ Hazardous Environment __ Other:	Required Reviewers: X Technical Reviewer __Building Manager __Radiological Control __ES&H X Quality Engineer X Other: Client X Other: Project Manager X Other: RPL Manager

Are One-Time Modifications Allowed to this Procedure?
x Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ____ **Yes or** _x_ **No**

FOR REVISIONS:

Is retraining to this procedure required? ___ Yes ___ No
Does the OJT package associated with this procedure require revision to reflect procedure changes? __ Yes__ No__ N/A

Approval:

	Signature	Date
Author	Gregg J. Lumetta	1/5/99
Technical Reviewer	Brian Lerch	1-11-99
RPL Manager	[signature]	1-12-99
Project Manager	D E Kurath	1/11/99
RPG QE	[signature]	1/12/99
BNFL	PS Lowrey	1/19/99

Applicability

This test plan is to be used to determine the effect of temperature on the solubility of entrained solids in the BNFL LAW samples. The work will be conducted in the SAL hot cells. The work will be conducted by Radiochemical Processing Group staff. This work is being done as part of the Technical Support to BNFL for Phase 1B project.

Test Objectives

Justification: This activity supports confirmation of the process sequence, equipment performance and design basis for the LAW entrained solids removal process. BNFL must complete research and testing activities conducted to confirm system design bases before March 1999.

Objective: Determine the components in solution at 30, 40, and 50°C and their concentrations. Infer from the solution composition the components dissolved in going from 30 to 40°C and from 40 to 50°C.

Definitions

BNFL	British Nuclear Fuels Ltd.
HDPE	High-density polyethylene
RPL	Radiochemical Processing Laboratory

Emergency Response

In the event of building audible alarms (e.g., fire or criticality) personnel should proceed in accordance with the RPL Building Emergency Procedure. If time permits, ensure that test materials are secured from spilling prior to exiting the area.

Quality Control

Quality assurance for work conducted under this Test Plan is governed by the Standards-Based Management System (SBMS). The quality control for each analysis will be established per Quality Assurance Plan MCS-033. MCS-033 specifies the minimum calibration and verification requirements for analytical systems, as well as batch processing quality control samples to monitor preparations (i.e., blanks, duplicates, matrix spikes, and laboratory control standards).

A work place copy of this document shall be present at the work location. Specific information regarding each test (e.g., sample numbers) will be recorded on the work place copy and kept as project records.

Hand written changes or corrections made to the work place copy will be made by means of a single line-out. Such changes or corrections shall be initialed and dated by the staff member making the change and by the cognizant scientist.

Equipment Description

A standard laboratory hot plate/magnetic stirrer will be used for this test. An aluminum heating block will be placed on the hot plate/stirrer to heat the sample. The apparatus will be equipped

with two thermocouples. One of the thermocouples will be connected to a temperature controller, while the other will be connected to an over-temperature shut-off device. The latter will be used to ensure the sample is not over heated, which could result in loss of sample.

Prerequisites

Staff performing the work must read and understand the entire test plan prior to beginning work.

The following are items that should be staged prior to start of the test.

30-mL HDPE bottle
20-mL HDPE vial (6)
Hot plate/stirrer
Aluminum heating block
Temperature controller with temperature read-out
Over-temperature shut-off device
0.45-µm nylon syringe filters (6)
5-mL syringes (6)
Adjustable 5-mL pipette
Boiling water bath
Small plastic bag

The temperature controller shall be calibrated by maintenance services. Record the following information regarding the temperature controller used.

Dual Thermocouple

Calibration ID: 02093 — 02899, 02900

Calibration Date: 1/12/99 — 1/99

Expiration Date: 1/2000 — 1/2001

Work Instructions

Note

Where practical, catch pans should be used when working with the tank waste samples, so that they can be recovered if spilled.

1. Prepare the sample vials according to the following table. All vials should be HDPE.

Sample ID[a]
AW101-SOL-30-1
AW101-SOL-30-2
AW101-SOL-40-1
AW101-SOL-40-2
AW101-SOL-50-1
AW101-SOL-50-2

(a) The prefix to the sample IDs should be the tank number; e.g. "AW101."

2. Label a 30-mL HDPE bottle as "AW101-SOL-TEST" (_____ = tank number) and place a magnetic stir bar in this bottle.

Place 25 mL of deionized water in the bottle and mark the liquid level. Empty the water from the bottle.

2/8/99 M.A.L. 3. Mix the stock LAW sample → AW-101 ST to give a homogeneous slurry

4. Transfer approximately 25 mL of the homogenized LAW slurry to AW101-SOL-TEST; use the 25-mL mark established in step 2 as a guide

Transferred the entire AW101 ST sample → Right at 25-mL mark.

5. Place AW101-SOL-TEST into an aluminum heating block thermostatted at 30°C

6. Stir the contents of AW101-SOL-TEST

7. Once the temperature has equilibrated at 30°C, stir the sample for at least 1 h

Start date/time: 2/8/99 / 13:30
Stop date/time: 2/8/99 / 15:00

8. Preheat two syringe/filter assemblies by placing them in a plastic bag and submersing the plastic bag with the syringe/filters into a boiling water bath

9. Withdraw a 2-mL aliquot of the slurry and filter into vial AW101-SOL-30-1

Filtered easily.

10. Withdraw a second 2-mL aliquot of the slurry and filter into vial AW101-SOL-30-2

11. Adjust the temperature of aluminum heating block assembly to 40°C

12. Once the temperature has equilibrated at 40°C, stir the sample for at least 1 h

2/9/99 M.A.L.

Start date/time: 2/9/99 / 15:20
Stop date/time: 2/9/99 / 8:15

13. Preheat two syringe/filter assemblies by placing them in a plastic bag and submersing the plastic bag with the syringe/filters into a boiling water bath

14. Withdraw a 2-mL aliquot of the slurry and filter into vial AW101-SOL-40-1

15. Withdraw a second 2-mL aliquot of the slurry and filter into vial AW101-SOL-40-2

16. Adjust the temperature of aluminum heating block assembly to 50°C

17. Once the temperature has equilibrated at 50°C, stir the sample for at least 1 h

Start date/time: 2/9/99 / 11:30 (time at which temp reached)
Stop date/time: 2/9/99 / 12:45

18. Preheat two syringe/filter assemblies by placing them in a plastic bag and submersing the plastic bag with the syringe/filters into a boiling water bath

19. Withdraw a 2-mL aliquot of the slurry and filter into vial AW101-SOL-50-1

20. Withdraw a second 2-mL aliquot of the slurry and filter into vial AW101-SOL-50-2

21. The samples collected during the test are to be submitted for the following analyses: IC(anions), TOC/TIC, acid digestion, ICP/AES, ICP-MS(Tc-99), Sr-90, total alpha, total uranium, and GEA. The cognizant scientist will prepare the required ASR.

Test completed at 12:51 on 2/9/99. M.J. Lumetta

Shielded Analytical Laboratory
Bench Sheet

Client: GREGG LAMETTA

WP Number: W48482

TI#/ASR: BNFL-TP-29953-7

Procedure: Determination of the Solubility of LAW Entrained Solids.

SAMPLE IDENTIFICATION

Test weights
20g = 19.9999
10g = 9.9998
5g = 4.9999

- Plastic bottles - - TARE -

AW 101-5L (with stir bar) - TEST = ~~30-1~~ 12.4570g
-30-1 = 6.6035g
-30-2 = 6.6305g
-40-1 = 6.5946g
-40-2 = 6.6210g
-50-1 = 6.6264g
-50-2 = 6.6320gr

2-8-99 transfered all solution in AW-101-ST into AW-101-5L.
2-9-99 completed AW 101-5L-TEST filtering into the 6 above labeled bottles @ 2mls ea.
2-10-99 first wash and filter complete, stirring with condenser for over night, AW 101-CL1
2-11-99 second wash and filter complete, stirring with condenser for over night, ~~AW 101~~,
2-12-99 third filter and wash complete, stiring will start Tues 16th.
2-16-99 Stir started. Temp @ 85°C.
2-17-99 fourth filter and wash complete, started next stir.
2-19-99 final H2O wash/filter complete, heating liquid to evaporate.
2-23-99 final weight of washed solids. = 0.0577g
2-25-99 Archived AW-101-AQ-30B, ...50B, ...70B, ...90B. → rack 4 slot 1,2,3,4
Transfered to clean vials to go out of cell - AW101-AQ-30A, ...50A, ...70A, ...90A, and AW101-50L-30A1, ...30A2, ...40A1, ...40A2, ...50A1, ...5A2.

M&TE: ✓ Cell 2 (360-06-01-016) Mettler AE160 Balance Other Denver Instrument Company A-16
(360-06-01-040) Due 2-99
___ Cell 5 (360-06-01-039) Mettler AT400 Balance
___ Bench (360-06-01-024) Sartorius Balance
___ Cell 5 (360-06-01-045) Toledo 3026 Balance

Analyst: K Kettan Date: 3-12-99

Reviewer: [signature] Date: 3/16/99

Shielded Analytical Laboratory
Bench Sheet

Client: Gregg Lumetta

WP Number:

TI#/ASR: BNFL-TP-29953-7

Procedure: Determination of the Solubility of LAW Entrained Solids.

SAMPLE IDENTIFICATION

2/28-99	Filtered AN 107 SOL 30-1 and 30-2 through a 1.2 micron filter, difficult.
3-3-99	Filtered AN 107 SOL 40-1 and 40-2 through a 1.2 micron filter, difficult. (2 filters each)
	Filtered AN 107 SOL 50-1 and 50-2 " " " " " " "
3-5-99	Added acid, evaporated. secured.
3-12-99	Redesolved residue, transfered solution out of cell to rm 511 in AW101-AQ-100C.
	final wt of AW101-AQ-100 = 16.7787.

M&TE: See pg 1 Cell 2 (360-06-01-016) Mettler AE160 Balance Other See pg 1

____ Cell 5 (360-06-01-039) Mettler AT400 Balance

____ Bench (360-06-01-024) Sartorius Balance

____ Cell 5 (360-06-01-045) Toledo 3026 Balance

Analyst:	Date:	Reviewer:	Date:
R. Tettan	3-12-99	Jack L. Steele	3/16/99

Appendix B. Raw Data

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

A0517
03/15/99
ASR5275

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	ICP/EQL @5 (ug/mL)	ICP/EQL @12.5 (ug/mL)	ICP/EQL @50 (ug/mL)	Filtrate, Wash Solutions MRQ (ug/mL)	12.5 99-1151-PB @1 Process Blank 3/15/99 (ug/mL)	50.0 99-1151 @2 AW101-SOL-30A1 3/15/99 (ug/mL)	50.0 99-1152 @2 AW101-SOL-30A2 3/15/99 (ug/mL)
0.015	Ag	0.8	1.9	7.5		--	[0.81]	[0.77]
0.060	Al	3.0	7.5	30.0	75.0	[7.3]	17,600	18,300
0.080	As	4.0	10.0	40.0		--	[12]	[12]
0.050	B	2.5	6.3	25.0		112	95.9	71.2
0.010	Ba	0.5	1.3	5.0	78.0	--	--	--
0.010	Be	0.5	1.3	5.0		--	[1.5]	[1.6]
0.100	Bi	5.0	12.5	50.0		--	--	--
0.100	Ca	5.0	12.5	50.0	150.0	--	[9.3]	[11]
0.015	Cd	0.8	1.9	7.5	75.0	--	[2.1]	[2.0]
0.100	Ce	5.0	12.5	50.0		--	--	--
0.025	Co	1.3	3.1	12.5	30.0	--	--	--
0.020	Cr	1.0	2.5	10.0	15.0	--	62.9	65.0
0.015	Cu	0.8	1.9	7.5	17.0	--	[1.6]	[1.6]
0.050	Dy	2.5	6.3	25.0		--	--	--
0.100	Eu	5.0	12.5	50.0		--	--	--
0.025	Fe	1.3	3.1	12.5	150.0	[0.39]	[3.5]	[3.5]
2.000	K	100.0	250.0	1000.0	75.0	--	24,400	25,600
0.025	La	1.3	3.1	12.5	35.0	--	--	--
0.005	Li	0.3	0.6	2.5		--	[0.57]	[0.44]
0.100	Mg	5.0	12.5	50.0	150.0	--	--	--
0.005	Mn	0.3	0.6	2.5	150.0	--	--	--
0.030	Mo	1.5	3.8	15.0	90.0	--	--	--
0.100	Na	5.0	12.5	50.0	75.0	117	143,000	145,000
0.100	Nd	5.0	12.5	50.0		--	--	--
0.030	Ni	1.5	3.8	15.0	30.0	--	[5.1]	[5.3]
0.100	P	5.0	12.5	50.0		--	344	358
0.060	Pb	3.0	7.5	30.0	300.0	--	38.9	42.7
0.300	Pd	15.0	37.5	150.0		--	--	--
0.300	Rh	15.0	37.5	150.0		--	--	--
0.075	Ru	3.8	9.4	37.5		--	[5.2]	[5.1]
0.050	Sb	2.5	6.3	25.0		--	--	--
0.050	Se	2.5	6.3	25.0		--	[5.7]	[5.3]
0.100	Si	5.0	12.5	50.0	170.0	119	264	202
1.000	Sn	50.0	125.0	500.0		--	[84]	[86]
0.005	Sr	0.3	0.6	2.5		--	--	--
0.500	Te	25.0	62.5	250.0		--	--	--
0.800	Th	40.0	100.0	400.0		--	--	--
0.005	Ti	0.3	0.6	2.5	17.0	[0.092]	--	--
0.250	Tl	12.5	31.3	125.0		--	--	--
2.000	U	100.0	250.0	1000.0	600.0	--	--	--
0.015	V	0.8	1.9	7.5		--	--	--
0.500	W	25.0	62.5	250.0		--	[74]	[76]
0.010	Y	0.5	1.3	5.0		--	--	--
0.020	Zn	1.0	2.5	10.0	16.5	[0.32]	[6.3]	[6.7]
0.025	Zr	1.3	3.1	12.5		--	[6.7]	[6.8]

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

A0517
03/15/99
ASR5275

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	50.0 99-1153 @2 AW101-SOL-40A1 3/15/99 (ug/mL)	50.0 99-1154 @2 AW101-SOL-40A2 3/15/99 (ug/mL)	50.0 99-1155 @2 AW101-SOL-50A1 3/15/99 (ug/mL)	50.0 99-1156 @2 AW101-SOL-50A2 3/15/99 (ug/mL)	12.5 99-1157 @1 AW101-AQ-30A 3/15/99 (ug/mL)
0.015	Ag	[0.84]	[0.81]	[0.84]	[0.82]	–
0.060	Al	18,600	18,600	19,200	18,700	1,080
0.080	As	[12]	[11]	[12]	[11]	–
0.050	B	98.0	89.8	89.2	94.7	31.3
0.010	Ba	–	–	–	–	–
0.010	Be	[1.6]	[1.6]	[1.7]	[1.6]	–
0.100	Bi	–	–	–	–	–
0.100	Ca	[11]	[11]	[12]	[10]	–
0.015	Cd	[2.1]	[2.0]	[2.1]	[2.0]	–
0.100	Ce	–	–	–	–	–
0.025	Co	–	–	–	–	–
0.020	Cr	67.5	67.4	70.6	68.8	64.4
0.015	Cu	[1.6]	[1.5]	[1.2]	[1.5]	–
0.050	Dy	–	–	–	–	–
0.100	Eu	–	–	–	–	–
0.025	Fe	[4.4]	[4.0]	[4.4]	[4.2]	–
2.000	K	26,000	26,000	26,700	26,000	1,590
0.025	La	–	–	–	–	–
0.005	Li	[0.51]	[0.39]	[0.48]	[0.40]	–
0.100	Mg	–	–	–	–	–
0.005	Mn	–	–	–	–	–
0.030	Mo	–	–	–	–	–
0.100	Na	146,000	146,000	147,000	146,000	12,000
0.100	Nd	–	–	–	–	–
0.030	Ni	[5.3]	[5.2]	[5.5]	[5.5]	[1.4]
0.100	P	361	357	372	364	[12]
0.060	Pb	48.6	40.0	40.7	42.0	[3.3]
0.300	Pd	–	–	–	–	–
0.300	Rh	–	–	–	–	–
0.075	Ru	[5.1]	[4.9]	[5.0]	[4.9]	–
0.050	Sb	–	–	–	–	–
0.050	Se	[5.5]	[5.5]	[4.7]	[5.0]	–
0.100	Si	269	274	248	269	79.8
1.000	Sn	[87]	[86]	[87]	[87]	–
0.005	Sr	–	–	–	–	–
0.500	Te	–	–	–	–	–
0.800	Th	–	–	–	–	–
0.005	Ti	–	–	–	–	–
0.250	Tl	–	–	–	–	–
2.000	U	–	–	–	–	–
0.015	V	–	–	–	–	–
0.500	W	[77]	[77]	[79]	[77]	–
0.010	Y	–	–	–	–	–
0.020	Zn	[6.6]	[6.6]	[7.0]	[6.7]	–
0.025	Zr	[7.0]	[7.0]	[7.2]	[7.1]	–

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

A0517
03/15/99
ASR5275

Det. Limit (ug/mL)	Multiplier= / Run Date= (Analyte)	12.5 99-1157-DUP @1 AW101-AQ-30A-DUP 3/15/99 (ug/mL)	5.0 99-1158 @1 AW101-AQ-50A 3/15/99 (ug/mL)	5.0 99-1159 @1 AW101-AQ-70A 3/15/99 (ug/mL)	5.0 99-1160 @1 AW101-AQ-90A 3/15/99 (ug/mL)
0.015	Ag	--	--	--	--
0.060	Al	1,100	89.6	49.6	38.5
0.080	As	--	--	--	--
0.050	B	29.8	27.5	27.4	27.5
0.010	Ba	--	[0.094]	[0.066]	[0.15]
0.010	Be	--	--	--	--
0.100	Bi	--	--	--	--
0.100	Ca	--	--	--	--
0.015	Cd	--	--	--	--
0.100	Ce	--	--	--	--
0.025	Co	--	--	--	--
0.020	Cr	65.6	5.36	1.72	[0.47]
0.015	Cu	--	--	--	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	--	[0.38]	[0.65]	[0.67]
2.000	K	1,640	[58]	--	--
0.025	La	--	--	--	--
0.005	Li	--	--	--	--
0.100	Mg	--	--	--	--
0.005	Mn	--	[0.093]	0.428	0.382
0.030	Mo	--	--	--	--
0.100	Na	12,300	783	283	243
0.100	Nd	--	--	--	--
0.030	Ni	[1.4]	--	--	--
0.100	P	[12]	[1.2]	[0.57]	--
0.060	Pb	--	[0.36]	--	--
0.300	Pd	--	--	--	--
0.300	Rh	--	--	--	--
0.075	Ru	--	--	--	--
0.050	Sb	--	--	--	--
0.050	Se	--	--	--	--
0.100	Si	68.5	62.9	72.4	61.2
1.000	Sn	--	--	--	--
0.005	Sr	--	--	--	--
0.500	Te	--	--	--	--
0.800	Th	--	--	--	--
0.005	Ti	--	--	[0.031]	[0.032]
0.250	Tl	--	--	--	--
2.000	U	--	--	--	--
0.015	V	--	--	--	--
0.500	W	--	--	--	--
0.010	Y	--	--	--	--
0.020	Zn	--	--	--	[0.25]
0.025	Zr	--	--	[0.27]	[0.25]

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory

SRPL 12 3/4/99 1.5 sieve e30 min

5-15-11

EXHIBIT I
PAGE 1 of 1

PNL-ALO-128 Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

Client name: Lumetta
Work Auth. Doc (WAD): ASR # 5275
Tank/Core/Project:
Special Instructions: for ASR 5275 states "No matrix spikes, LCS or blank spikes"

Work package number: Multiple
Project number: 29953
PNL QA plan:
PNL Impact level:
Prep. lab (SAL/SRPL/other): SRPL
Preparation batch number:

	ACL Sample ID	ACL order number or Client sample ID	Vial Identifier	Sample Volume (ml)	Spike added Volume (ml)	Spike added Weight (g)	Final solution Volume (ml)	Process Factor (1)
1	PB-1151	—		2mL H2O	NA	NA	25mL	
2	99-1151	AW101-SOL-30A1		1 mL				
3	99-1152	AW101-SOL-30A2						
4	99-1153	AW101-SOL-40A1						
5	99-1154	AW101-SOL-40A2						
6	99-1155	AW101-SOL-50A1						
7	99-1156	AW101-SOL-50A2						
8	99-1157	AW101-AQ-30A		2mL				
9	99-1157Dup	AW101-AQ-30A						
10	99-1158	AW101-AQ-50A		5mL				
11	99-1159	AW101-AQ-70A						
12	99-1160	AW101-AQ-90A						
13								
14								

Analyst's sample preparation comments: Pipet checks: #112498 #L0577DN @ 5mL
Balance # 360-06-01-026

1.0013g	4.9833g	
1.0016g	4.9782g	
1.0017g	5.0012g	
1.0001g	4.9951g	
0.9971g	5.0020g	

Spike source:
PNL spike ID number:
Anal. balance M&TE:
Sample filtered (yes/no):

(1) Process factor = Final volume (ml) /Sample volume (ml)

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Analyst/Date: R.K. Berg 5/4/99 Reviewer/Date:

Rev. 2.0 7-28-95 JMR

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329/4 File

Date March 10, 1999

To Mike Urie

From James Bramson

Subject ICP/MS Analysis of Submitted Samples
(ACL #99-1151 through 99-1160)

Pursuant to your request, the 11 samples that you submitted for analysis were analyzed by ICPMS for ^{99}Tc. The results of this analysis are reported on the attached page.

An Amersham ^{99}Tc standard was used to generate the calibration curve and an independent Amersham ^{99}Tc standard was used as the continuing calibration verification (CCV) standard. The 1% high-purity nitric acid solution used to dilute the standards and samples was used as a reagent blank. The samples were diluted an extra 5x (99-1159, 99-1160) and 20x (all others) from the dilutions provided. The results include your dilutions and are reported in ng/ml (ppb) of the original sample. Unless otherwise specified, the overall uncertainty of the values is conservatively estimated at ±10%, and is based on the precision between consecutive analytical runs as well as the accuracy of the CCV standard results.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc. The fingerprint we're seeing for Ru is obviously not natural, and is consistent with that observed in previous tank waste analyses. Ru counts, corrected for sample dilution, are provided for your information.

If you have any questions regarding this analysis, feel free to call me at 372-0624 or Tom Farmer at 372-0700.

J Brown 3/10/99

Lumetta Tc-99 Samples

March 10, 1999

The results are reported in ng/ml (ppb) of original sample.
The uncertainty of the results is estimated at ±10%.

Sample Number	Client Number	ICP/MS Number	Tc-99 ng/ml	Ru ratio 101/102 (*0.541)	†Ru-101 ng/ml
1%HNO3		9309a1	<0.05		
1%HNO3		9309a10	<0.05		
1%HNO3		9309a25	<0.05		
PB-1151		9309a11	<0.05	1.5217	3
99-1151	AW101-SOL-30A1	9309a21	6030	1.1917	1700
99-1151 + spike	AW101-SOL-30A1	9309a24	8740		
Spike Recovery			**108%**		
99-1152	AW101-SOL-30A2	9309a19	6230	1.1788	1800
99-1152 Dup.	AW101-SOL-30A2	9309a23	6210	1.2040	1700
99-1153	AW101-SOL-40A1	9309a20	6320	1.1136	1700
99-1154	AW101-SOL-40A2	9309a22	6230	1.1630	1700
99-1155	AW101-SOL-50A1	9309a17	6520	1.1806	1800
99-1156	AW101-SOL-50A2	9309a18	6400	1.1421	
99-1157	AW101-AQ-30A	9309a15	1380	1.1868	150
99-1157 Dupe	AW101-AQ-30A	9309a16	1390	1.0951	150
99-1158	AW101-AQ-50A	9309a14	149	1.2497	15
99-1159	AW101-AQ-70A	9309a13	47.4	1.0146	0.8
99-1160	AW101-AQ-90A	9309a12	23.1	1.6620	0.5
1ppb Tc-99		9309a7	0.977		
10ppb Tc-99		9309a26	10.3		
10ppm Co		9309a5	<0.05		

* Natural $^{101}Ru/^{102}Ru$ ratio.
†Based on response from yttrium.

Tc-99 0.017 Ci/g

$$\frac{ng}{mL} \cdot \frac{g}{10^9\ ng} \cdot \frac{0.017\ Ci}{g} \cdot \frac{10^6\ \mu Ci}{Ci}$$

DATA REVIEW

Reviewed by: [signature]

Date: 11 Mar 99 Pages: ____

AW101 Tank Liquids and Wash Solutions (ASR 5275)
Radiochemistry Analytical Results

Sample Preparation

Tank liquid and wash solution samples were analyzed from tank AW101. The samples were acid digested according to procedure PNL-ALO-128 in the laboratory prior to analysis. Radiochemistry results are shown on the attached table along with 1-sigma total uncertainties. All results are reported on a uCi per ml of liquid. Samples labeled "duplicate" are independent analyses from separate aliquots of starting material in the hot cell; those labeled "replicate" are separate aliquots analyzed in the laboratory.

Gamma Energy Analysis

The acid digested samples were directly gamma counted following procedure PNL-ALO-450. Most of the gamma emission from these samples is from Cs-137. The only other detectable gamma emitters were Co-60 and Cs-134. The prep blank had a negligible amount of Cs-137.. All of the duplicate results agree within the expected uncertainties. Since gamma analyses do not involve chemical separations, no sample spiking is performed. Due to the high level of Cs-137 in these samples, it was not possible to detect all of the other analytes at the requested Minimum Reportable Quantity values. Detection limits are thus reported for Eu-154, Eu-155, and Am-241.

Gross Alpha

For gross alpha measurements, aliquots of the digested samples were evaporated on planchets for counting following procedures PNL-ALO-420 and 421. Weak alpha activity was only detectable in two of the wash solutions. All of the other samples had detection limits well below the requested MRQ values. Sample and blank spike recoveries were acceptable. No alpha activity was found in either the prep blank or the lab blank.

Strontium-90

The Sr-90 analyses were conducted according to procedures PNL-ALO-476, 484, and 450 using a Sr-85 tracer to monitor the chemical yields. All of the samples had detectable levels of Sr-90. Sr-90 was not detected in the hot cell blanks. The blank and sample spike recoveries were acceptable. Duplicate results were in acceptable agreement considering the uncertainties on the measurements.

Uranium

Uranium was measured directly in the digested samples by kinetic phosphorescence following procedure PNL-ALO-4014. Uranium was detectable in all of the samples with concentrations ranging from 1-4 ug/ml. A negligible amount of uranium was seen in the prep blank; no uranium was detected in the lab blank. No uranium was detectable in the instrument blanks. The duplicate samples were in good agreement. All of the instrument check standards came out between 99% and 102%.

T R Greenwood
3-30-99

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

99-1151 Rev. 1
3/30/99

Client : Lumetta

Cognizant Scientist: L R Greenwood Date : 3/30/99

Concur : T Trang-Le Date : 3/30/99

Measured Activities (uCi/ml)

ALO ID Client ID	Alpha Error %	Sr-90 Error %	Uranium ug/ml Error %	Co-60 Error %	Cs-134 Error %	Cs-137 Error %	Am-241 Error %	Eu-154 Error %	Eu-155 Error %
99-1151PB AW101-SOL-30A1	<4.E-5	<1.E-4	6.96E-5 3%	<9.E-6	<8.E-6	1.55E-5 29%	<5.E-5	<3.E-5	<3.E-5
99-1151 AW101-SOL-30A1	<6.E-3	9.49E-1 14%	2.73E+0 2%	<4.E-3	5.48E-2 10%	2.55E+2 2%	<2.E-1	<1.E-2	<2.E-1
99-1151 Rep AW101-SOL-30A1	<6.E-3								
99-1152 AW101-SOL-30A2	<6.E-3	4.00E-1 30%	2.80E+0 2%	<4.E-3	5.94E-2 9%	2.64E+2 2%	<2.E-1	<9.E-3	<2.E-1
99-1153 AW101-SOL-40A1	<7.E-3	5.19E-1 24%	3.00E+0 2%	<3.E-3	5.68E-2 10%	2.67E+2 2%	<2.E-1	<1.E-2	<2.E-1
99-1154 AW101-SOL-40A2	<6.E-3	6.96E-1 18%	2.98E+0 2%	<4.E-3	5.73E-2 9%	2.64E+2 2%	<2.E-1	<1.E-2	<2.E-1
99-1155 AW101-SOL-50A1	<6.E-3	5.34E-1 23%	3.15E+0 2%	<2.E-3	5.77E-2 7%	2.76E+2 2%	<7.E-2	<1.E-2	<7.E-2
99-1156 AW101-SOL-50A2	<8.E-3	3.52E-1 34%	3.08E+0 2%	<2.E-3	6.09E-2 7%	2.72E+2 2%	<7.E-2	<9.E-3	<7.E-2
99-1157 AW101-AQ-30A	<3.E-4	3.44E-2 18%	3.00E+0 4%	<4.E-4	3.03E-3 16%	1.69E+1 2%	<2.E-2	<2.E-3	<1.E-2
99-1157 DUP AW101-AQ-30A	<3.E-4	4.16E-2 15%	3.05E+0 4%	<4.E-4	3.18E-3 18%	1.71E+1 2%	<2.E-2	<2.E-3	<1.E-2
RPD		19%	2%		5%	1%			
99-1158 AW101-AQ-50A	<5.E-5	1.89E-2 8%	1.19E+0 4%	<7.E-5	2.38E-4 20%	1.14E+0 2%	<3.E-3	<2.E-4	<2.E-3
99-1158 Rep AW101-AQ-50A		1.60E-2 9%							
99-1159 AW101-AQ-70A	2.70E-4 10%	7.19E-2 3%	4.45E+0 4%	4.60E-5 17%	6.67E-5 19%	3.15E-1 2%	<7.E-4	<8.E-5	<5.E-4
99-1160 AW101-AQ-90A	2.52E-4 11%	6.85E-2 3%	4.16E+0 4%	<6.E-5	<7.E-5	1.88E-1 2%	<1.E-3	<2.E-4	<8.E-4
Matrix Spike	93%	117%							
Reagent Spike	88%	104%							
Blank	<3.E-6	<1.E-4	<-1.78E-5						
Before Run UL 283-e			100%						
R-283-c			99%						
Mid Run UL -283-f			101%						
R-283-d			102%						
Post Run UL 283-e			99%						
R-283-d			102%						

Lumetta, Gregg J
From: Greenwood, Larry R
Sent: Tuesday, March 30, 1999 4:00 PM
To: Lumetta, Gregg J
Subject: RE: Additional Info for ASR#5275

Gregg - If you look at the report, the 1 sigma total propagated uncertainties are rather large for the Sr-90 determinations. For example, 30A1 is +/-14% and 30A2 is +/-30%. The two values just about overlap at the 2 sigma limit. All of the other cases look like they would overlap at the 1 sigma limit. The reason for the relatively high uncertainty is that the Sr-90 activity was lower than a weak beta background caused by the Sr-85 tracer that we added. (We count Sr-85 by gamma emission; however, Sr-85 does have a very weak atomic electron emission.) We tried to guess at how much tracer to add based on the apparent high beta activity in the samples. Unfortunately, the Cs-137 activity accounted for virtually all of the beta and the Sr-90 activity was lower than we guessed. There is no way that we could have known this in advance since we did not measure total beta for you (for comparison with the GEA, for example). If the statistical scatter is too high, then we could rerun the samples with a lower Sr-85 tracer activity. (If you want to consider this option, the cost would be about $3500) As you can see, the hotter samples (50A, 70A, and 90A) have much lower uncertainties since these samples are much hotter than the tracer background.

Larry Greenwood *Ph: 509-376-6918 Fax: 509-372-2156*
mailto:larry.greenwood@pnl.gov

----Original Message----
From: Lumetta, Gregg J
Sent: Tuesday, March 30, 1999 3:45 PM
To: Greenwood, Larry R
Subject: RE: Additional Info for ASR#5275

Thanks, Larry.

There seems to be considerable scatter in the Sr-90 data. For example, samples AW101-SOL-30A1 and -30A2, should essentially be identical; yet the reported concentrations for Sr-90 are 0.949 and 0.400 µCi/mL, respectively. Likewise, -40A1 and -40A2 differ (0.519 versus 0.696) as does -50A1 and -50A2 (0.534 versus 0.352).

Any thoughts on this matter?

Gregg

-----Original Message-----
From: **Greenwood, Larry R**
Sent: Tuesday, March 30, 1999 9:43 AM
To: Lumetta, Gregg J
Cc: Urie, Michael W; Fiskum, Sandra K
Subject: RE: Additional Info for ASR#5275

Gregg - I have also prepared a brief narrative for these samples. << File: 5275 Narrative.doc >>

Larry Greenwood *Ph: 509-376-6918 Fax: 509-372-2156*
mailto:larry.greenwood@pnl.gov

----Original Message----
From: Greenwood, Larry R
Sent: Tuesday, March 30, 1999 8:51 AM

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

WO/Project: W48481&W48482/29953
Client: G. Lumetta

ACL Nmbr(s): 99-1151 through 99-1160

Client ID: AW101 SOL and AW101 AQ series

ASR Nmbr 5275

Total Samples: 10 liquids

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography" (IC).

Analyst: MJ Steele

Analysis Date: March 30-31, 1999 and Reruns April 12-13, 1999

See Chemical Measurement Center 98620: IC File for Calibration and Maintenance Records.

M&TE Number: IC instrument -- WD25214
Mettler AT400 Balance – Cal. No. 360-06-01-031

Analyst: MJ Steele 4/27/99

Approval: [signature] 4/27/99

Final Results:

Ten liquid samples were analyzed by ion chromatography (IC) for inorganic anions as specified in ASR 5275. The liquid samples were diluted 5-fold to 12.25-fold during the preparation of the samples prior to distribution to the IC workstation, and were diluted at the IC workstation up to 200-fold to ensure that all anions were within the calibration range. The samples were initially analyzed on March 30-31, 1999. From this run, the verification standards for many analytes were below the 90% recovery acceptance criteria. Therefore the samples were reanalyzed on April 12-13, 1999. Only results from the final analysis run are provided in this report. The results from the initial analysis run are included in the data package for information only.

Based on client communications the nitrate result for AW101-SOL-40A2 appears to be about a factor of two higher than expected. The only other analyte in this sample at a high enough concentration to provide reliable results nitrite, and the nitrite for sample AW101-SOL-40A2 is only slightly higher than sample AW101-SOL-40A1. To provide sufficient sample for all analyses requested, AW101-SOL samples had to be diluted 10-12 fold; it is possible that the small volume sample was contaminated during the initial dilution. Both the initial run (which failed QC) and the final run measured nitrate above 200,000 µg/ml.

The results for the samples from the April 12-13, 1999 run are presented in the table below.

SAMPLE ID	Client ID	Dil Factor	F µg/ml	Cl µg/ml	NO2 µg/ml	Br µg/ml	NO3 µg/ml	PO4 µg/ml	SO4 µg/ml	C204 µg/ml
99-1151 PB	PROCESS BLANK	5	<1.4	<1.4	<2.8	<1.4	<2.8	<2.8	<2.8	<2.8
	% RECOVERY		91	97	100	97	96	94	95	98
99-1151	AW101-SOL-30A1	12.25	1,300	3,600	69,400	<600	118,000	<1200	<1200	<1200
99-1152	AW101-SOL-30A2	10	1,300	4,100	62,300	<500	120,000	<1000	<1000	<1000
	% RECOVERY		91	115	110	105	111	100	103	107
99-1153 #1	AW101-SOL-40A1	12.25	1,600	4,400	75,300	<600	122,000	<1200	<1200	<1200
99-1153 #1 REPLICATE	AW101-SOL-40A1	12.25	1,400	5,000	80,000	<300	154,000	<613	1,752	<613
	RPD (%)		15	14	6	n/a	24	n/a	n/a	n/a
99-1153 #2	AW101-SOL-40A1	12.25	1,100	3,000	65,400	<300	124,000	1,400	1,400	<600
99-1153 #2 REPLICATE	AW101-SOL-40A1	12.25	1,200	3,800	64,600	<300	125,000	1,400	1,600	<600
	RPD (%)		6	22	1	n/a	0	4	11	n/a
99-1154	AW101-SOL-40A2	12.25	1,600	4,200	72,700	<600	227,000	<1200	<1200	<1200
99-1155	AW101-SOL-50A1	10	1,600	4,100	65,200	<500	126,000	<1000	<1000	<1000
99-1156	AW101-SOL-50A2	10	1,500	4,100	63,500	<500	122,000	<1000	<1000	<1000
99-1157	AW101-AQ-30A	5	100	250	3,800	<25	7,300	<50	120	6,400
99-1158	AW101-AQ-50A	5	6.0	11	170	<1.4	360	<2.8	6.5	210
99-1159	AW101-AQ-70A	5	<1.4	3.0	8.0	<1.4	34	<2.8	<2.8	<2.8
99-1160	AW101-AQ-90A	5	<1.4	2.5	<2.8	<1.4	2.7	<2.8	<2.8	<2.8
	% RECOVERY		98	110	108	107	103	101	104	86

RPD = Relative Percent Difference (between sample and duplicate)

Q.C. Comments:

Following are results of quality control checks performed during IC analyses. In general, quality control checks met the requirements of the governing QA Plan, MCS-033.

Working Blank Spike/Process Blank Spike: Process Blank Spike recoveries ranged from 91% to 100%, well within the acceptance criteria of 75% to 125%.

Matrix Spiked Sample: The matrix spike recovery for samples AW101-SOL-30A2 and AW101-AQ-90A ranged from 86% to 115%. Again, this is well within the acceptance criteria of 75% to 125%.

Duplicate: No duplicates were provided. However, the laboratory-dilution of sample AW101-SOL-40A1 was analyzed in replicate (i.e., two different analysis injections) at the IC workstation from two different IC workstation dilutions. Two replicate analyses failed the acceptance criteria of a Relative Percent Difference less than 20%; nitrate on IC dilution #1 and chloride on IC dilution #2. Based on QC performance of matrix spikes and verification standards, no explanation can be offered for the poor precision on the one nitrate from IC dilution #1. However, there are significant interference peaks between the fluoride and nitrite retention times than can account for the poor precision of the chloride results, since chloride peak baselines are difficult to establish.

System Blank/Processing Blanks: No anions were detected above reportable concentrations in the system blanks or in the processing/dilution blank.

Quality Control Calibration Verification Check Standards: Five mid-range verification standards were analyzed throughout the analysis run. For all reported results, the concentrations of all analytes of interest were recovered within the governing QA Plan acceptance criteria of ± 10% for the verification standard.

Notes:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis.

2) The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.

3) Routine precision and bias is typically ± 15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions. Sample-specific precision and bias may be determined on each sample if required.

Battelle
Putting Technology To Work

Project No. 29953

Internal Distribution

Date April 6, 1999 File/LB

To G. Lumetta

From M. Urie

Subject Carbon Analysis Results for AW-101 SOL and AW-101 AQ Samples

The analysis of the AW-101-SOL and AW-101-AQ samples submitted under ASR 5275 was done by the hot persulfate wet oxidation method, PNL-ALO-381, rev. 1. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95 °C for TOC, all on the same weighed sample, with TC being the sum of the TIC and TOC.

The samples were analyzed on April 1, 1999 and Table 1 below shows the results, rounded to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank.

Due to the limited quantity of original sample available and the number of different analyses requested, the sample were diluted to provide enough volume for each of the analyses. All results are corrected for preparative dilutions and analysis dilutions, and are reported in microgram of carbon per milliliter of original sample.

QC Narrative

The TIC standard is calcium carbonate and TOC standard is α-Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC).

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type. The QC system calibration standards were all within acceptance criteria, with the average recovery being 93.9% for TIC and 97.1% for TOC. The calibration blanks were acceptable, averaging 16.7 µgC for TIC and 33.7 µgC for TOC.

The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spike recovery from sample 99-1160 106% for TIC and 103% for TOC, well within the acceptance criteria of 75% to 125%. The precision, estimated by the RPD (Relative Percent Difference) between duplicates, could not be measured since the duplicate contained carbon less than 5 times the estimated quantitation limit.

Some results are reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data.

Table 1: TIC, TOC, and TC Results

ALO Number	Sample ID	Vol ml	Prep Dilution Factor	TIC* µgC/ml	TIC RPD	TOC* µgC/ml	TOC RPD	TC* µgC/ml	TC RPD
99-1151 PB	PROCESS BLANK	2.00	5.00	<25		<40		< 65**	
99-1151	AW101-SOL-30A1	1.00	12.25	2760		1900		4660	
99-1152	AW101-SOL-30A2	1.00	10.00	2960		1940		4900	
99-1153	AW101-SOL-40A1	1.00	12.25	3040		2010		5050	
99-1154	AW101-SOL-40A2	1.00	12.25	2940		1960		4900	
99-1155	AW101-SOL-50A1	1.00	10.00	3170		2010		5180	
99-1156	AW101-SOL-50A2	1.00	10.00	2730		2030		4760	
99-1157	AW101-AQ-30A	0.50	5.00	410		1900		2310	
99-1158	AW101-AQ-50A	0.50	5.00	190		<170		360**	
99-1159	AW101-AQ-70A	1.00	5.00	120		<80		200**	
99-1160	AW101-AQ-90A	1.00	5.00	120		<80		200**	
99-1160 Dup	AW101-AQ-90A Dup	1.00	5.00	120	n/a	<80	n/a	200**	n/a
99-1160 Spike	AW101-AQ-90A Spike (%rec)	1.00	5.00	106%		103%		105%	

* Corrected for laboratory dilution performed prior to analysis
** Maximum TC (i.e., results calculated as if "< values" present in the sample)
RPD = Relative Percent Difference between sample and duplicate (n/a = not calculated since results <5xMDL)

Approve: _____ 4-6-99

Archive Information:

Files: C124-P-701.doc, C124-701.xls ASR: 5275

Appendix C. Calculations

Battelle
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ENGINEERING WORKSHEET

Prepared By:	Date:	Project:
S.L. Lumetta	3/31/99	29953

Title/Subject: AW 101 LAW Entrained Solids Solubility Versus Temperature

At each temperature examined (30, 40, and 50°C), two samples were taken for analysis. In most cases, single analyses of these samples were performed, in which case the mean value was determined by:

$$\text{Mean} = \frac{V1 + V2}{2} \qquad (1)$$

where V1 is the value determined for one sample and V2 is the value determined for the other. The standard deviation from the mean was determined using the following equation:

$$\sigma = \sqrt{\frac{n\sum x_i^2 - (\sum x_i)^2}{n(n-1)}} \qquad (2)$$

n = # data points
x_i = measured value

or in this case:

$$\sigma = \sqrt{\frac{2(V1^2+V2^2) - (V1+V2)^2}{2}} \qquad (3)$$

Example: Chromium Concentration at 30°C

Sample AW101-SOL-30A1 → 62.9 mg/mL (V1)
Sample AW101-SOL-30A2 → 65.0 mg/mL (V2)

$$\text{Mean} = \frac{62.9 + 65.0}{2} = 64.0 \text{ mg/mL}$$

$$\sigma = \sqrt{\frac{2(62.9^2 + 65.0^2) - (62.9 + 65.0)^2}{2}} = 1.5 \text{ mg/mL}$$

Battelle
Pacific Northwest Laboratories

ENGINEERING WORKSHEET

Prepared By:	Date:	Project:
D.J. Temmerman	3/31/99	29953

Title/Subject: AW101 Entrained Solids Solubility Versus Temperature

For the Tc-99 analysis, sample AW101-SOL-30A2 was run in duplicate. In this case the mean value was taken as "V2".

i.e.,

AW101-SOL-30A1 → 6030 ng/mL (V1)

AW101-SOL-30A2 → 6230
AW101-SOL-30A2 Dup → 6210
→ $\frac{6230+6210}{2}$ = 6220 ng/mL (V2)

Note: The Tc-99 values were converted to units of μCi/mL as follows.

$$\left(6030 \frac{ng}{mL}\right)\left(\frac{g}{10^9 ng}\right)\cdot\left(\frac{0.017 Ci}{g}\right)\left(\frac{10^6 \mu Ci}{Ci}\right) = 0.103 \mu Ci/mL$$

(0.017 Ci/g = specific activity of ^{99}Tc)

$$\left(6220 \frac{ng}{mL}\right) \cdot \text{(same factors)} = 0.106 \mu Ci/mL$$

∴

$$\text{Mean} = (0.103 + 0.106/2) = 0.104 \mu Ci\ ^{99}Tc/mL$$

$$\sigma = \sqrt{\frac{2(0.103^2 + 0.106^2) - (0.103 + 0.106)^2}{2}} = 0.002 \mu Ci/mL$$

A Microsoft Excel® spreadsheet was used to execute the above described calculations. The results are compiled in the attached tables.

See tables 1 → 4

Appendix D. Statistical Analysis of the Data

STATISTICAL ANALYSES

Statistical analyses were performed on the concentration data from sampling the liquid portion of the AW-101 sample twice each at temperatures of 30, 40 and 50°C. Attention was focused on concentrations of Cs-137, Sr-90, and Tc-99 (in units of μCi/mL) and Al, Cr, Na, U, TOC, TIC, Cl, F, and NO_3 (in units of μg/mL).

D.1 Data Plots

A *scatterplot matrix* plot of the concentration data and temperature values is given in Figure D.1. This plot shows all possible pairwise scatter plots of the different variables on one page. The diagonal of the scatterplot matrix lists the variable labels, which apply to the horizontal axes of the plots in a column, and the vertical axes of the plots in a row. The scatter plots above the diagonal are just reflected images of the scatter plots below the diagonal. Scatterplot matrix plots are useful for seeing correlations or relationships among variables, and for identifying possible outlying or "different" data points.

All but the last row and column of the scatterplot matrix in Figure D.1 contain scatter plots of two component concentrations. Most of the individual scatter plots show moderate to very strong positive linear correlations between component concentrations. For example, the pairwise correlation between concentrations of Cs-137 and Tc-99 is very strong, as is the correlation between Cr and U. Several of the scatter plots show moderate correlation, which may be due to subsampling or analytical variations or uncertainties. Still other scatter plots suggest the possibility of outlying (different) data points. Most notable in this regard are points in the plots of components versus TIC, Cl, F, and NO_3. The concentrations for these components and the steps of their generation should be examined in light of these plots to ascertain whether there may be incorrect data.

The last row and column of the scatterplot matrix in Figure D.1 contain scatter plots of component concentrations versus temperature. In these plots as well as the concentration plots, different plotting symbols were used for the three temperature values to help display the effect of temperature on component concentrations. For several components, there appears to be strong linear relationships between concentration and temperature, with concentration increasing as temperature increases. For TIC and F, there are different/outlying data that reduce the apparent strength of linear relationships. In the case of F, the questionable point is at 50°C. All questionable data points should be assessed and appropriate action taken.

D.2 Regression Analyses

To quantitatively assess the nature of linear relationships between component concentration and temperature, equations of the form

$$Concentration = a + b\ Temperature \qquad (D.1)$$

were fit to the data by ordinary (unweighted) least squares regression methods using the Minitab (1998) software package. In (D.1), a is the estimated intercept, and b is the estimated slope. Because two subsamples were taken at each of three temperatures, there are six data points to fit (D.1) for each component. The two concentration values per temperature provide for obtaining a pooled (combined) three degree-of-freedom estimate of experimental uncertainty. In this case, the experimental uncertainty

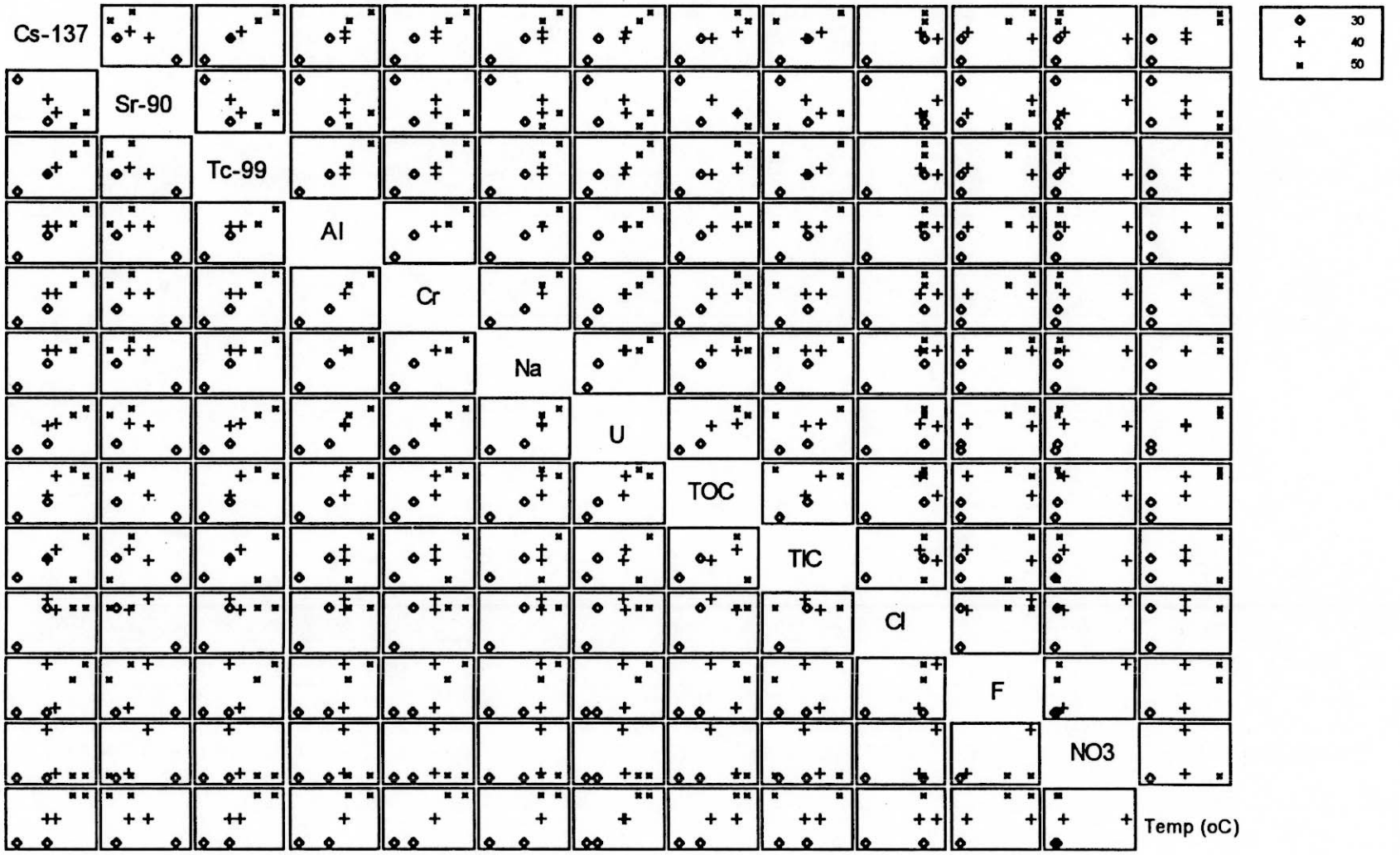

Figure D.1. Scatterplot Matrix of AW-101 Component Concentration and Sample Temperature Data

comprises subsampling and analytical uncertainties. This leaves one degree-of-freedom for assessing the lack-of-fit of the linear equation in (D.1). Technically, a quadratic polynomial equation in temperature could be fit instead of the linear equation, but then there would be no basis for assessing lack-of-fit. It should be kept in mind that six data points per equation provides a limited basis for estimating experimental uncertainty and assessing lack-of-fit.

The results of the linear regression analyses are summarized in Table D.1. For each AW-101 component, Table D.1 shows the estimated intercept and slope of the fitted linear equation (D.1). Also shown is the confidence level at which the slope is statistically different from zero, the R^2 and s "goodness of fit" statistics, and an indication whether the linear equation has a statistically significant lack-of-fit. The terms and concepts are explained in footnotes of Table D.1. Of the AW-101 components considered, Cs-137, Tc-99, Al, Cr, Na, U, TOC, and F had slopes statistically different from zero with confidence level of 90% or greater. The R^2 values for these components ranged from 0.583 for F to 0.936 for U. The closer an R^2 value is to 1.00, the better the fit of (D.1) to the data for that component. The R^2 values for some of the components are considerably below 1.00 because of the large experimental uncertainty in the replicate data at each temperature. A fitted regression equation cannot account for replicate uncertainties, which lowers the achievable maximum value of R^2.

The regression results confirm what is seen from the scatter plots in the last row and column of Figure D.1. Those plots show there are strong linear relationships between concentration and temperature for Cs-137, Tc-99, Al, Cr, Na, U, and TOC. The lesser strength of the linear relationship for F could be due to an outlying data point. The slope for TIC not being statistically different from zero could be due to the different/outlying point at 50°C mentioned in the earlier discussion of Figure D.1.

The regression results for NO_3 require some discussion. The regression was performed without the outlying second observation at 40°C. The resulting fitted line does not have a slope statistically different from zero. However, there is a statistically significant lack-of-fit. This suggests that a quadratic rather than a linear relationship may be more appropriate. However, there is no basis for asessing the goodness of fit of a quadratic polynomial, since it would exactly fit the means of the NO_3 concentrations at the three temperatures. The fact that the evidence for a quadratic relationship relies on the single remaining observation at 40°C being higher than the mean concentrations at 30°C and 50°C suggests considerable caution be used regarding this result.

D.3 Discussion of Statistical Results

In the regression work of Section D.1, the statistical ability to detect slopes different from zero is limited by having only two replicate samples/measurements at each temperature, and having relatively large variability in the two values. Hence, any slopes found as statistically different from zero probably are. However, slopes not found as statistically different from zero might have been found different from zero if there had been more replicate subsamples/analyses, or if the variability in subsamples/analyses were smaller.

The higher pairwise correlations between many of the component concentrations leads to finding slopes statistically different from zero for highly correlated components. Sometimes people mistakenly think that significant differences in more components is somehow stronger evidence of significant differences, but in the presence of high correlations (in component concentrations in this case), it has to turn out that way. Hence, it is not necessarily stronger evidence.

Finally, it is up to subject matter experts to decide whether statistically significant differences are of practical significance.

PNWD-2466
BNFL-RPT-004, Rev. 0

LETTER REPORT

AW-101 Entrained Solids – Solubility Versus Temperature

Gregg J. Lumetta
Ralph C. Lettau
Greg F. Piepel

August 1999

Prepared for BNFL, Inc. under
Project 29952/29953
Battelle, Richland, Washington, 99352

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Contents

Introduction

BNFL, Inc. (BNFL) is under contract with the U.S. Department of Energy, River Protection Project (DOE-RPP) to design, construct, and operate facilities for the treatment of wastes stored in the single-shell and double-shell tanks at the Hanford Site, Richland Washington. BNFL has contracted with Battelle Pacific Northwest Division to conduct tests to verify and validate the BNFL waste treatment process. The DOE-RPP has provided samples from tanks 241-AW-101, 241-AN-107, 241-C-104, and 241-C-106 to BNFL for this purpose.

This report describes the results of a test conducted by Battelle to assess the solubility of the solids entrained in the diluted AW-101 low-activity waste (LAW) sample. BNFL requested Battelle to dilute the AW-101 sample using de-ionized water to mimic expected plant operating conditions. BNFL further requested Battelle to assess the solubility of the solids present in the diluted AW-101 sample versus temperature conditions of 30, 40, and 50°C. BNFL requested these tests to assess the composition of the LAW supernatant and solids versus expected plant-operating conditions. The work was conducted according to test plan BNFL-TP-29953-7, Rev. 0, *Determination of the Solubility of LAW Entrained Solids*. The test went according to plan, with no deviations from the test plan.

Personnel

The Battelle personnel and their responsibilities in performing this test are given below.

Staff Member	Responsibilities
G.J. Lumetta	Cognizant scientist. Prepared test plan and designed experiment. Supervised performance of the test. Prepared analytical service request. Interpreted data and reported results.
R.C. Lettau	Hot cell technician. Performed test.
M.W. Urie	Managed chemical and radiochemical analytical work.
B.M. Rapko	Technical reviewer.
K.P. Brooks	Task Leader.
G.F. Piepel	Statistical analysis of the data.

Experimental

Sample Description. The sample used in this test was labeled as AW-101 ST. The homogenization, dilution, caustic adjustment, and sub-sampling of the as-received AW-101 sample were described by Urie 1999. The total volume of sample AW-101 ST was 25 mL and it contained approximately 2 mL of settled solids.

Apparatus. The apparatus used consisted of an aluminum heating block placed on a hot plate/stirrer. The hot plate/stirrer was modified so that separate power could be applied to the heating and stirring functions. This allowed for continuous stirring, while the hot plate was powered by a temperature controller. The temperature controller used was a J-KEM Model 270 (J-KEM

Electronics, Inc., St. Louis, MO). This temperature controller consists of two separate circuits. One is the temperature control circuit, while the other serves as an over-temperature device, which shuts down the system if a preset temperature is exceeded. The set point for the over-temperature circuit was set at 60°C for this test. A dual K-type thermocouple (model number CASS-116G-12-DUAL, Omega Engineering, Stamford, CT) was used to provide inputs to the temperature controller and over-temperature circuits. Both the J-KEM Model 270 and the dual thermocouple were calibrated before use. The aluminum heating block contained two wells. A vial containing water was placed in one of the wells, with the thermocouple wedged between this vial and the aluminum block. The vial containing the sample was placed in the other well.

Procedure.[a] The sample in AW-101 ST was mixed by swirling. The homogenized slurry was then transferred to a 30-mL high density polyethylene (HDPE) vial (this vial also contained a Teflon®-coated magnetic stir bar). The sample was heated and stirred at 30 ± 2 °C for 1.5 h. Two aliquots (2-mL each) were taken for analysis. Each aliquot was immediately filtered through a 0.45-µm nylon syringe filter that had been preheated by immersion in a boiling water bath. The filter was preheated to avoid precipitation during the filtration step. The temperature was increased to 40 ± 2 °C and the sample was stirred for 16.75 h.[b] The mixture was sampled in the same manner as described above. The temperature was increased to 50 ± 2 °C and the sample was stirred for 1.25 h. Again, the mixture was sampled in the same manner as described above. The filtered samples were subjected to the following analytical procedures: IC(anions), TOC/TIC, acid digestion, ICP/AES, ICP-MS(Tc-99), Sr-90, total alpha, total uranium, and GEA.

Results

Tables 1, 2, and 3 present the concentrations of various waste components at 30, 40, and 50°C, respectively. Table 4 shows the changes in the concentrations at 40 and 50°C relative to those at 30°C. Appendix D discusses a graphical analysis of the data, as well as linear regression results of fitting component concentrations versus temperature. The following discussion is organized according to the following types of components: 1) radionuclides, 2) bulk metals and carbon, and 3) anions.

Radionuclides. The data suggest that the ^{137}Cs concentration increased slightly with temperature. Increases of 2.3 and 5.6% in the ^{137}Cs concentration occurred in going from 30 to 40°C and from 30 to 50°C, respectively. Nearly identical increases were seen in the ^{99}Tc concentrations. The increases in ^{137}Cs and ^{99}Tc concentrations from 30 to 50°C were statistically significant (see Table 4). Linear regressions of ^{137}Cs and ^{99}Tc concentrations versus temperature had statistically significant positive slopes (see Appendix D).

On the other hand, the ^{90}Sr concentrations appeared to decrease with increasing temperature. However there is considerable scatter in the ^{90}Sr data; the standard deviations range from 20 – 58% of the mean ^{90}Sr concentration values at each temperature. Thus, the indicated changes in the ^{90}Sr

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[a] The test plan and the associated procedural notes are included as Appendix A to this report.

[b] The test plan required the AW-101 sample to be maintained at temperature for at least 1 hour before sampling. For convenience, the sample was maintained at 40°C overnight. It should be noted that this test was not designed to address the kinetics of dissolution. Kinetics could potentially be important regarding the phenomena investigated here, but separate testing would be required to address this issue.

concentrations are not statistically significant (see Table 4). Similarly, the linear regression of ^{90}Sr concentration versus temperature had a slope that was not statistically different from zero (see Appendix D). The reason for the relatively high uncertainty in ^{90}Sr concentrations was the relatively high background caused by the ^{85}Sr tracer that was added in the analytical procedure to monitor Sr recovery.

All the transuranic elements (alpha emitters) were below the detection limits. Likewise, the europium isotopes $^{154,155}Eu$ were not detected. It should be noted that the detection limits for the Eu isotopes were somewhat high because of the strong ^{137}Cs activity in the sample.

Bulk Metals and Carbon. Most of the metals analyzed showed slight concentration increases with increasing temperature. Most notable are the increases seen for Al, Cr, and U. Increases in these three components are statistically significant based on the analyses presented in Table 4 and Appendix D. The Fe concentration increased approximately 20% when the temperature was raised to 40 or 50°C, with the increases assessed to be statistically significant (see Table 4). However, Fe was near the detection limit so that there is significant experimental uncertainty associated with this result. The concentrations of K, Na, Ni, P, and Zr also displayed statistically significantly increases with increasing temperature.

The total organic carbon (TOC) concentration in the AW-101 solution also increased slightly with increasing temperature, with the increase being statistically significant (see Table 4 and Appendix D). The average total inorganic carbon (TIC) concentration increased when the temperature was raised from 30 to 40°C, but the TIC concentration did not increase further when the temperature was raised to 50°C. The changes from 30 to 40°C and 30 to 50°C are not statistically significant, because of the experimental uncertainties in the TIC measurements (see Table 4).

Anions. The data suggest that the F^- concentration increased with temperature, with the increase being statistically significant. Increases of 12.5 and 19.2% in the F^- concentration occurred in going from 30 to 40°C and from 30 to 50°C, respectively (see Table 4). The linear regression of F^- concentration versus temperature also had a statistically significant positive slope (see Appendix D). The data also suggest the average Cl^- concentration increased when the temperature was raised from 30 to 40°C, although the increase was not statistically significant (see Table 4). The Cl^- concentration did not increase further when the temperature was raised to 50°C. Statistical analyses of these data suggest the F^- and Cl^- concentration increases should be considered with caution as there is considerable scatter in the data. Sulfate and phosphate ions were below the detection limits of the ion chromatograph. Assuming phosphate ion is the dominant form of P in solution, the behavior of PO_4^{3-} can be deduced from the ICP data as discussed above.

In determining the concentration of NO_3^-, there was a significant discrepancy between the duplicate analyses for the solution taken at 40°C. In particular, the nitrate concentration value obtained for sample AW101-SOL-40A2 was about twice that obtained for sample AW101-SOL-40A1. Furthermore, the value of 131,250 μg/mL obtained for sample AW101-SOL-40A1 was more in line with those obtained for the solution at 30 and 50°C. This strongly suggests that the value reported for AW101-SOL-40A2 is in error. Perhaps this error was caused by nitrate contamination of the sample, or a dilution error. Using the value of 131,250 μg/mL at 40°C, the data suggest a 10% increase in the nitrate concentration when the temperature is raised from 30 to 40°C. However, there was a decrease in the nitrate concentration when the temperature was raised from 40 to 50°C. The linear regression of NO_3^- concentration versus temperature (omitting the concentration value

for AW101-SOL-40A2 as an outlier) had a statistically significant lack of fit (see Appendix D). This result suggests that a quadratic rather than linear relationship may be more appropriate. However, the limited nature of the data (especially after omitting the outlier) raises the question whether the decrease between 40 and 50°C is significant.

Conclusions

The data are limited because they are based on a single AW-101 sample, from which was obtained two subsamples/analyses at each of three temperatures. Further, the data for some AW-101 components are subject to considerable uncertainty. However, there does appear to be an overall trend for the concentrations of certain AW-101 waste components (e.g., ^{137}Cs, ^{99}Tc, Al, Cr, K, Na, Ni, P, U, Zr, TOC, F^-, and NO_3^-) to increase with increasing temperature. Typical increases were on the order of 2 to 5% for each 10°C increase, although a fewer larger increases were seen for some components. Because the sample bottle was sealed during the course of the experiment, evaporation in not a likely cause for the observed concentration increases.

Reference

Urie, M.W. et al. 1999. *Inorganic and Radiochemical Analysis of AW-10 and AN-107 "Diluted Feed" Materials*, PNWD-2463, Battelle Pacific Northwest Division, Richland, Washington.

Table 1. AW-101 Component Concentrations in Solution at 30°C.[a]

Analyte	Concentration at 30°C			Std. Dev.
	AW101-SOL-30A1	AW101-SOL-30A2	Mean	
Cesium-137	255	264	260	6
Strontium-90	0.949	0.400	0.675	0.388
Technetium-99	0.103	0.106	0.104	0.002
Americium-241	< 6E-03	< 6E-03	< 6E-03	--
Europium-154	< 1E-02	< 9E-03	< 9E-03	--
Europium-155	< 2E-01	< 2E-01	< 2E-01	--
Total Alpha	< 6E-03	< 6E-03	< 6E-03	--
Ag	(0.81)	(0.77)	(0.79)	0.03
Al	17600	18300	17950	495
Ba	< 5.0	< 5.0	< 5.0	--
Ca	(9.3)	(11.0)	(10.2)	1.2
Cd	(2.1)	(2.0)	(2.1)	0.1
Co	< 12.5	< 12.5	< 12.5	--
Cr	62.9	65.0	64.0	1.5
Cu	(1.6)	(1.6)	(1.6)	0.0
Fe[b]	(3.5)	(3.5)	(3.5)	0.0
K	24400	25600	25000	849
La	< 12.5	< 12.5	< 12.5	--
Mg	< 50	< 50	< 50	--
Mn	< 2.5	< 2.5	< 2.5	--
Mo	< 15	< 15	< 15	--
Na	143000	145000	144000	1414
Ni	(5.1)	(5.3)	(5.2)	0.1
P	344	358	351	10
Pb	38.9	42.7	40.8	2.7
Si[c]	264	202	233	44
Ti	< 2.5	< 2.5	< 2.5	--
U	2.73	2.80	2.77	0.05
Zn[d]	(6.3)	(6.7)	(6.5)	0.3
Zr	(6.7)	(6.8)	(6.8)	0.1
TOC	1900	1940	1920	28
TIC	2760	2960	2860	141
Cl^-	3600	4100	3850	354
F^-	1300	1300	1300	0
NO_3^-	118000	120000	119000	1414
SO_4^{2-}	< 1200	< 1000	< 1200	--
PO_4^{3-}	< 1200	< 1000	< 1200	--

(a) Concentrations for radionuclides are in units of µCi/mL; all other components are in units of µg/mL. Values in parentheses are near the analytical detection limit.
(b) The process blank had a relatively high Fe content of 0.4 µg/mL.
(c) The process blank had a relatively high Si content of 119 µg/mL.
(d) The process blank had a relatively high Zn content of 0.3 µg/mL.

Table 2. AW-101 Component Concentrations in Solution at 40°C.[a]

Analyte	Concentration at 40°C			
	AW101-SOL-40A1	AW101-SOL-40A2	Mean	Std. Dev.
Cesium-137	267	264	266	2
Strontium-90	0.519	0.696	0.608	0.125
Technetium-99	0.107	0.106	0.107	0.001
Americium-241	< 7E-03	< 6E-03	< 6E-03	--
Europium-154	< 1E-02	< 1E-02	< 1E-02	--
Europium-155	< 2E-02	< 2E-02	< 2E-02	--
Total Alpha	< 7E-03	< 6E-03	< 6E-03	--
Ag	(0.84)	(0.81)	(0.83)	0.02
Al	18600	18600	18600	0
Ba	< 5.0	< 5.0	< 5.0	--
Ca	(11.0)	(11.0)	(11.0)	0.0
Cd	(2.1)	(2.0)	(2.1)	0.1
Co	< 12.5	< 12.5	< 12.5	--
Cr	67.5	67.4	67.5	0.1
Cu	(1.6)	(1.5)	(1.6)	0.1
Fe[b]	(4.4)	(4.0)	(4.2)	0.3
K	26000	26000	26000	0
La	< 12.5	< 12.5	< 12.5	--
Mg	< 50	< 50	< 50	--
Mn	< 2.5	< 2.5	< 2.5	--
Mo	< 15	< 15	< 15	--
Na	146000	146000	146000	0
Ni	(5.3)	(5.2)	(5.3)	0.1
P	361	357	359	3
Pb	48.6	40	44.3	6.1
Si[c]	269	274	272	4
Ti	< 2.5	< 2.5	< 2.5	--
U	3.00	2.98	2.99	0.01
Zn[d]	(6.6)	(6.6)	(6.6)	0.0
Zr	(7.0)	(7.0)	(7.0)	0.0
TOC	2010	1960	1985	35
TIC	3040	2940	2990	71
Cl^-	4050	4200	4125	106
F^-	1325	1600	1462.5	194
NO_3^-	131250	227000	179125	67705
SO_4^{2-}	< 1600	< 1200	< 1600	--
PO_4^{3-}	< 1400	< 1200	< 1400	--

(a) Concentrations for radionuclides are in units of µCi/mL; all other components are in units of µg/mL. Values in parentheses are within 10 times the analytical detection limit.
(b) The process blank had a relatively high Fe content of 0.4 µg/mL.
(c) The process blank had a relatively high Si content of 119 µg/mL.
(d) The process blank had a relatively high Zn content of 0.3 µg/mL.

Table 3. AW-101 Component Concentrations in Solution at 50°C.[a]

Analyte	Concentration at 50°C			Std. Dev.
	AW101-SOL-50A1	AW101-SOL-50A2	Mean	
Cesium-137	276	272	274	3
Strontium-90	0.534	0.352	0.443	0.129
Technetium-99	0.111	0.109	0.110	0.001
Americium-241	< 6E-03	< 8E-03	< 6E-03	--
Europium-154	< 1E-02	< 9E-03	< 9E-03	--
Europium-155	< 7E-02	< 7E-02	< 7E-02	--
Total Alpha	< 6E-03	< 8E-03	< 6E-03	--
Ag	(0.84)	(0.82)	(0.83)	0.01
Al	19200	18700	18950	354
Ba	< 5.0	< 5.0	< 5.0	--
Ca	(12.0)	(10.0)	(11.0)	1.4
Cd	(2.1)	(2.0)	(2.1)	0.1
Co	< 12.5	< 12.5	< 12.5	--
Cr	70.6	68.8	69.7	1.3
Cu	(1.2)	(1.5)	(1.4)	0.2
Fe[b]	(4.4)	(4.2)	(4.3)	0.1
K	26700	26000	26350	495
La	< 12.5	< 12.5	< 12.5	--
Mg	< 50	< 50	< 50	--
Mn	< 2.5	< 2.5	< 2.5	--
Mo	< 15	< 15	< 15	--
Na	147000	146000	146500	707
Ni	(5.5)	(5.5)	(5.5)	0.0
P	372	364	368	6
Pb	40.7	42.0	41.4	0.9
Si[c]	248	269	259	15
Ti	< 2.5	< 2.5	< 2.5	--
U	3.15	3.08	3.12	0.05
Zn[d]	(7.0)	(6.7)	(6.9)	0.2
Zr	(7.2)	(7.1)	(7.2)	0.1
TOC	2010	2030	2020	14
TIC	3170	2730	2950	311
Cl^-	4100	4100	4100	0
F^-	1600	1500	1550	71
NO_3^-	126000	122000	124000	2828
SO_4^{2-}	< 1000	< 1000	< 1000	--
PO_4^{3-}	< 1000	< 1000	< 1000	--

(a) Concentrations for radionuclides are in units of µCi/mL; all other components are in units of µg/mL. Values in parentheses are within 10 times the analytical detection limit.
(b) The process blank had a relatively high Fe content of 0.4 µg/mL.
(c) The process blank had a relatively high Si content of 119 µg/mL.
(d) The process blank had a relatively high Zn content of 0.3 µg/mL.

Table 4. Concentration Changes Relative to 30°C[a]

Analyte	Change, %[b] 40°C	Change, %[b] 50°C	%RSD[c]	SD of % Change[c]	%Change / SD of % Change[c] 40°C	%Change / SD of % Change[c] 50°C
Cesium-137	2.3	**5.6**	1.6	2.3	1.02	**2.46**
Strontium-90	-9.9	-34.3	39.1	55.3	-0.18	-0.62
Technetium-99	2.4	**5.5**	1.4	2.0	1.20	**2.69**
Americium-241	(d)	(d)	(d)	(d)	(d)	(d)
Europium-154	(d)	(d)	(d)	(d)	(d)	(d)
Europium-155	(d)	(d)	(d)	(d)	(d)	(d)
Total Alpha	(d)	(d)	(d)	(d)	(d)	(d)
Ag	(4.4)	(5.1)	2.7	3.9	1.15	1.31
Al	3.6	**5.6**	1.9	2.7	1.33	**2.05**
Ba	(d)	(d)	(d)	(d)	(d)	(d)
Ca	8.4	8.4	10.1	14.3	0.59	0.59
Cd	(0.0)	(0.0)	3.4	4.9	0.00	0.00
Co	(d)	(d)	(d)	(d)	(d)	(d)
Cr	**5.5**	**9.0**	1.7	2.4	**2.27**	**3.73**
Cu	-(3.1)	-(15.6)	9.4	13.4	-0.23	-1.17
Fe	**(20.0)**	**(22.9)**	4.3	6.1	**3.27**	**3.74**
K	4.0	**5.4**	2.2	3.2	1.26	**1.70**
La	(d)	(d)	(d)	(d)	(d)	(d)
Mg	(d)	(d)	(d)	(d)	(d)	(d)
Mn	(d)	(d)	(d)	(d)	(d)	(d)
Mo	(d)	(d)	(d)	(d)	(d)	(d)
Na	1.4	**1.7**	0.6	0.9	1.55	**1.94**
Ni	(1.0)	**(5.8)**	1.8	2.5	0.39	**2.33**
P	2.3	**4.8**	1.9	2.7	0.84	**1.79**
Pb	8.6	1.3	8.9	12.6	0.68	0.11
Si[e]	16.5	10.9	11.4	16.1	1.03	0.68
Ti	(d)	(d)	(d)	(d)	(d)	(d)
U	**8.1**	**12.7**	1.4	2.0	**4.08**	**6.35**
Zn	(1.5)	(5.4)	3.1	4.4	0.35	1.23
Zr	**(3.7)**	**(5.9)**	0.8	1.2	**3.15**	**5.04**
TOC	**3.4**	**5.2**	1.4	2.0	**1.72**	**2.64**
TIC	4.5	3.1	6.9	9.7	0.47	0.32
Cl^-	7.1	6.5	5.5	7.8	0.92	0.83
F^-	12.5	**19.2**	8.1	11.5	1.09	**1.68**
NO_3^-	**10.3**[f]	4.2	1.82[f]	2.57[f]	**4.01**[f]	1.63[f]
SO_4^{2-}	(d)	(d)	(d)	(d)	(d)	(d)
PO_4^{3-}	(d)	(d)	(d)	(d)	(d)	(d)

(a) Values in parentheses are for analytes that were within 10 times the analytical detection limit.

(b) The percent change is given by: $\%Change = 100*(C_T - C_{30})/C_{30}$, where C_T is the average concentration at temperature T (40 or 50°C) and C_{30} is the average concentration at 30°C.

(c) %RSD is the percent relative standard deviation, obtained as the root mean square of the %RSD values at 30°C, 40°C, and 50°C. SD of % Change is the standard deviation of the % Change values at 40°C and 50°C, both relative to 30°C. It is computed as Sqrt(2)*%RSD. % Change/SD of % Change is the number of standard deviations the % Change value is from zero. Assuming a statistical t-distribution with 3 degrees of freedom, % Change/SD of % Change values must be larger than 1.64 to be significant at the 90% (one-sided) confidence level. Such values, and their corresponding % Change values, are shown in boldface.

(d) Analyte not detected.

(e) The values for Si should be veiwed with caution because of the high process blank.

(f) Value obtained using a value of 131250 µg NO_3^-/mL at 40°C.

Appendix A. Test Plan

Appendix B. Raw Data

Appendix C. Calculations

Appendix D. Statistical Analysis of the Data

Table D.1. Results of Linear Regressions of AW-101 Component Concentrations Versus Temperature

AW-101 [a] Component	Intercept	Slope	Confidence Level for Slope ≠ 0 [b]	R^2 [c]	s [d]	LOF [e]
Cs-137	237.333	0.725	98.3%	0.792	3.71	no
Sr-90	1.0380	-0.0116	65.6%	0.224	0.216	no
Tc-99	0.0960	0.000275	98.3%	0.796	0.00139	no
Al	16500.00	50.00	96.6%	0.714	316.2	no
Cr	55.533	0.288	99.5%	0.884	1.043	no
Na	140500.0	125.0	95.0%	0.658	901.4	no
U	2.2567	0.0175	99.8%	0.936	0.0459	no
TOC	1775.00	5.00	98.3%	0.797	25.25	no
TIC	2753.30	4.500	35.4%	0.058	181.3	no
Cl	3525.00	12.50	71.3%	0.273	203.9	no
F	937.50	12.50	92.3%	0.583	105.7	no
NO_3 [f]	113450.0	250.0	58.1%	0.225	5356	yes

(a) Components with slopes significantly different from zero with statistical confidence 90% or greater are shown in bold. The practical significance of the slopes must be assessed based on subject matter knowledge.

(b) The confidence level (in %) that the slope is significantly different from zero.

(c) R^2 is the proportion of variation in response (concentration, for these data) values accounted for by the fitted equation. Theoretically, $0 \le R^2 \le 1$, but because there are replicate data points at each temperature, the maximum R^2 can achieve is less than one. This result is due to the fact that a fitted equation cannot account for replicate variability. Hence the more replicate variability there is, the lower R^2 will be regardless of how well the fitted equation approximates the relationship.

(d) Assuming the fitted equation adequately approximates the data, s is an estimate of the experimental uncertainty standard deviation. For these data, s has the concentration units of the corresponding component.

(e) This column contains results of a statistical significance test (at the 90% confidence level) on whether the fitted equation had a lack-of-fit (LOF).

(f) The 227,000 concentration for the second subsample/analysis of NO_3 at 40°C was excluded as an outlier for purposes of the regression analysis reported here.

Reference

Minitab (1998), *Minitab Statistical Software, Release 12*, Minitab, Inc., State College, PA.